भारतीय मानक
Indian Standard

IS 8050 : 2018

अल्फा पिकोलीन — विशिष्टि

(पहला पुनरीक्षण)

Alpha Picoline — Specification

(First Revision)

UDC No 667·271: 547·821 411

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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 9

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by Petroleum, Coal and Related Products Division Council.

Alpha picoline is used in the manufacture of picolinic acid, 2-vinyl pyridine, 2-pyridylethanol, 2 beta-methoxy ethyl pyridine and pyridine 2-aldehyde. It also finds use in dyestuff industry as a solvent.

This standard was first published in 1976 stipulating the requirements and methods of test for two grades of alpha picoline, namely, Grade 1 and grade 2 having alpha picoline content 98.0 percent and 96.5 percent, respectively.

In the present revision (first revision) the Committee observed that alpha picoline Grade 2 with purity 96.5 percent is neither in demand nor produced, hence deleted Grade 2. Subsequently, the requirement of alpha picoline has been modified to 99.0 percent, *Min*. The gas chromatographic method of analysis of alpha picoline has also been upgraded.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALPHA PICOLINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for Alpha picoline.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on the subject are encouraged to investigate the possibilities of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)
2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
4905 : 2015	Random sampling and randomization procedures (*first revision*)
5298 : 2013	Method for determination of distillation range and distillation yield (*second revision*)

3 REQUIREMENTS

3.1 Description

The material shall be clear to yellow liquid having a strong characteristic odour.

3.2 Solubility

The material shall be soluble in in water in all proportions.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in this standard.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in mild steel drums. The gaskets for the bungs shall be of high density polythene.

4.2 Marking

4.2.1 Each container shall be suitably marked with the following information:

a) Name and grade of the material;
b) Net mass of the material in the container;
c) Name of the manufacturer and his recognized trade-mark, if any;
d) Batch number or lot number, in code or otherwise, and
e) The symbol given in Fig. 5 of IS 1260 (Part 1).

4.2.2 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5 SAMPLING

The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex D.

Table 1 Requirements for Alpha Picoline
(*Clause* 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Alpha picoline content, percent by mass, *Min*	99.0	A
ii)	Boiling range at 760 mm Hg	2 to 97 percent by volume shall distil between a range of 128.0 to 131.0°C	B
iii)	Moisture content, percent by mass, *Max*	0.25	C

6 TEST METHODS

6.1 Tests shall be conducted as prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No*. (i)]

DETERMINATION OF ALPHA PICOLINE CONTENT

A-1 OUTLINE OF METHOD

The content of alpha picoline along with other components is determined by gas chromatography. The chromatographic conditions given here are for guidance only.

A-2 APPARATUS

A-2.1 Gas Chromatograph — Equipped with flame-ionization detector using capillary column.

A-2.2 Data Acquisition system — A system capable of acquiring chromatographic data and integrating chromatographic peaks.

A-2.3 Column—CP-WAX-52CB, 60 m long, 0.25 mm ID, Film thickness df = 0.25 µm.

A-3 REAGENTS

A-3.1 Certified Reference/Working Standard of Pyridine

A-3.2 Certified Reference/Working Standard of Alpha Picoline

A-3.3 Certified Reference/Working Standard of Gamma Picoline

A-3.4 Certified Reference/Working Standard of 2,6-Lutidine

A-3.5 Internal Standard — Pentadecane, Purity more than 99 percent.

NOTE — Working standard is prepared against Certified Reference Standard.

A-4 PROCEDURE

A-4.1 Operating Parameters of Gas Chromatograph

A-4.1.1 *Injector Temperature* : 240°C.

A-4.1.2 *Detector Temperature*: 250°C.

A-4.1.3 *Carrier Flow* (*He*) : 1.5 ml/min.

A-4.1.4 *Split Ratio* : 1: 100.

A-4.1.5 *Make up Flow* (N_2) : 20 ml/min.

A-4.1.6 *Oven Temperature*-1: 75°C for 18 min.

A-4.1.7 *Oven Temperature*-2 : 250°C for 1 min.

A-4.1.8 *Programming Rate* : 10°C /min.

A-4.1.9 *Total Run Time* : 35 min.

A-4.1.10 *Range* : 20.

A-4.1.11 *Attenuation*: –3.

A-4.2 Standard and Test Sample Preparation

A-4.2.1 *For Chromatographic Purity*

Standard and test sample, inject 0.2 µl.

A-4.2.2 *Preparation of Internal Standard (Pentadecane) Solution — 10 mg/ml*

Weigh and transfer accurately 1 000 mg of pentadecane in a 100 ml volumetric flask. Add methanol to dissolve and dilute the content to the volume. If required sonicate till stock solution becomes clear and preserve in a cool place. Pipette out 4.0 ml of the above solution into another 100 ml volumetric flask and dilute to the mark

with methanol. Final concentration of pentadecane is about 0.4 mg/ml

A-4.2.3 *Preparation of Standard Solution for Calibration*

Weigh accurately 970 ± 0.5 mg of working reference sample Alpha picoline and 10 mg each working reference sample of Pyridine, Gamma picoline and 2,6-Lutidine in a clean and dry vial and mix thoroughly.

Weigh accurately about 200 ± 0.5 mg of the above synthetic mixture into another 10 ml volumetric flask and accurately add 5.0 ml of internal standard solution by pipette. Dissolve and mix the contents.

A-4.2.4 *Test Sample Preparation*

Weigh accurately about 200 ± 0.5 mg of sample of Alpha Picoline in a clean and dry 10 ml volumetric flask and dilute to 5 ml of internal standard solution by pipette. Dissolve the contents and mix thoroughly.

A-4.3 Methods

Condition the column at 250°C for 30 min. Allow the gas chromatograph to equilibrate at 75°C and obtain a steady baseline before proceeding with analysis. Following is the recommended sequence for analysis and evaluating suitability:

a) Inject 1.0 µl of blank (Methanol) — Single injection.
b) Inject 1.0 µl of calibration solution — Duplicate injection.
c) Inject 0.2 µl (neat) each of standard and test sample — Single injection.

Ensure the system suitability criteria as given below before proceeding further and determine the response factor from the calibration chromatogram nearest to 0.001:

Sl No.	*Method Performance Parameter*	*Criteria*
i)	Diluent blank has no significant interfering peaks, if any subtract the blank peak area from those of the standard and the sample	
ii)	Tailing factor for all analytes	≤ 2.5
iii)	Resolution between of all impurities	≥ 1.5

A-4.4 Calculation

Alpha picoline content is calculated by corrected normalization method.

A-4.4.1 The area/mass (*A*/*M*) ratio by dividing the area of each peak by its mass as under:

Component	*Mass, Percent(M)*	*Area*	*A/M Ratio*
Pentadecane	1.0	A_1	$A_1/1.0 = K$
Pyridine	1.0	A_2	$A_2/97.0 = L$
Alpha picoline	97.0	A_3	$A_3/1.0 = M$
Gamma picoline	1.0	A_4	$A_4/1.0 = N$
2,6-Lutidine	1.0	A_5	$A_5/1.0 = O$

A-4.4.2 *Actual Mass Percent in Calibration Solution*

Set arbitrarily internal standard response factor to 1.0 and find response factor of other components as follows:

Component	*Slope*	*Response Factor*
Pentadecane	K/K	1.000 0
Pyridine	K/L	Put the value obtained
Alpha picoline	K/M	Put the value obtained
Gamma picoline	K/N	Put the value obtained
2,6-Lutidine	K/O	Put the value obtained

A-4.4.3 Multiply the areas by their response factor to get the true areas of the peaks. Add up the area to get total true area and calculate as per following:

Component 'n' in the sample (percent by mass) =

$$\frac{A_n \times (100 - M)}{A_t}$$

where

A_n = peak area of component '*n*',
m = percent of water in the sample, and
A_t = total true peak area.

A-4.4.4 *Typical Retention Time (RT) of Interest Components in Alpha Picoline*

Sl No.	*Component Name*	*Approx.RT (in min)*	*Type of Component*
i)	Pyridine	10.5	Impurity
ii)	Alpha picoline	12.2	Finished Product
iii)	2,6-Lutidine	13.5	Impurity
iv)	Gamma picoline	17.8	Impurity
v)	Pentadecane	25.6	Internal Standard

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF BOILING RANGE

B-1 PROCEDURE

B-1.1 Determine the boiling range by the procedure prescribed in IS 5298 applying the following corrections.

B-1.1.1 *Correction of Thermometer Reading*

B-1.1.1.1 *Error of scale*

In all thermometer readings, make the corrections as described on the certificate of the instrument.

B-1.1.1.2 *Correction of barometric pressure*

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If, however, the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall be corrected as follows:

a) For every 27 mm above 760 mm Hg, subtract 1°C from the specified temperature; and

b) For every 27 mm below 760 mm Hg, add 1°C to the specified temperature.

NOTE — These corrections are valid only for pressure above 700 mm Hg.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF MOISTURE CONTENT

C-1 GENERAL

Moisture is determined by the Karl Fischer method.

C-2 PROCEDURE

Weigh accurately 20 g of the material and determine the moisture content by the procedure given in IS 2362.

ANNEX D

(*Clause* 5)

SAMPLING OF ALPHA PICOLINE

D-1 GENERAL REQUIREMENTS FOR SAMPLING

D -1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

D -1.2 The sampling instrument shall be clean and dry.

D-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

D-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

D-1.6 The sample containers shall be of such a size that they are almost three-fourth filled by the sample.

D-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given in **4.2**.

D-2 SCALE OF SAMPLING

D-2.1 Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

D-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

D-2.3 The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected from Lots of Different Sizes
(*Clause* D-2.3)

Lot Size '*N*'	Sample Size '*n*'
(1)	(2)
3 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 and above	9

D-2.4 The containers shall be chosen at random from the lot with the help of a suitable random number table. Reference may be made to IS 4905 for guidance to random selection procedures.

D-3 TEST SAMPLE AND REFEREE SAMPLE

D-3.1 From each of the containers selected as in **D-2.3**, draw with the help of a sampling bottle a representative portion of the material from different parts of the container. Out of this portion from each container equal quantity of the material shall be taken and thoroughly mixed to form a composite sample of about 1 500 ml. This composite sample shall be thoroughly mixed and divided into three equal portions, one for the purchaser, another for the supplier and the third for the referee.

D-3.2 The remaining portion corresponding to each of the selected containers as in **D-2.3**, shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

D-3.3 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **D-1.7**.

D-3.4 The referee samples consisting of a composite sample and a set of *n* individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

D-4 TESTS

D-4.1 Tests for Alpha picoline content and moisture shall be conducted on individual samples.

D-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

D-5 CRITERIA FOR CONFORMITY

D-5.1 For Individual Samples

The lot shall be declared as conforming to the requirements of Alpha picoline content and moisture if each of the test results on the individual samples satisfies the corresponding requirement of the test.

D-5.2 For Composite Sample

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results shall satisfy the relevant requirements given in **3** and Table 1.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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